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Polycyclic Aromatic Hydrocarbons on the Inner Surfaces of Copper Tubing

by
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MAY 1984

NAVAL WEAPONS CENTER
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FOREWORD

Copper tubing and pipe are used to heat, cool, and transport gases and liquids for many applications. The contamination of this tubing with toxic, mobile materials could have implications for public health and could be a contamination problem in scientific instrumentation. This report discusses the finding of significant quantities of polycyclic aromatic hydrocarbons (PAHs) on the inner surfaces of copper tubing as supplied by the manufacturers. The work covered in this report was performed during July and August 1982 with Pollution Abatement Research Funds, Program Element Number 62765N, NAVSEA Task Area Number SF65559 under sponsorship of Dr. George Young.

This work was reviewed for technical accuracy by D. W. Moore and D. A. Fine.

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Research Department
15 March 1984

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(U) Polycyclic aromatic hydrocarbons (PAHs) have been determined by gas chromatography/mass spectrometry (GC/MS) to be present on the inner surfaces of copper tubing as the tubing was supplied by the manufacturers. The PAHs probably exist as by-products of the manufacturing process. Concentrations of PAHs as high as 0.17 mg/m of tubing were found. A summary of the various PAHs found in three samples of copper tubing is included with this report. 

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INTRODUCTION

Ongoing research at the Naval Weapons Center involves tests aimed at determining air pollutants which may be released into the environment by operations which are peculiar to the military (References 1 and 2). After performing one of these tests, we discovered that one of our traps was heavily contaminated with polycyclic aromatic hydrocarbons (PAHs). The source of the contamination was traced to a section of new copper tubing which we had used as a gas sample probe.

Since copper is such a good conductor of heat, copper tubing is usually used in applications where a maximum heat transfer is desired, e.g., refrigerators, radiators, air conditioners, and cold water fountains. The malleability of copper makes the tubing an excellent choice for many gas or liquid lines used in scientific equipment.

PAHs are formed when free radicals combine during the combustion or pyrolysis of organic materials (Reference 3). Many of these compounds have been identified as carcinogens or mutagens. One particular PAH, benzo(a)pyrene, has been identified as one of the most prevalent environmental carcinogens to which man is exposed (Reference 3). The presence of carcinogenic PAHs in drinking water is of concern to health scientists (Reference 4). Since copper tubing is used to transport and cool water, it could be an important source of PAH contamination.

Aside from possible health aspects, contamination of scientific instrumentation which utilizes copper tubing for carrier lines could be a significant problem. In the case of eximer lasers, for instance, contaminated purge lines could result in the deposition of PAHs on the laser optics, causing a reduced efficiency of the instrument. Contaminated carrier lines in gas chromatography could have a disastrous effect on the interpretation of resulting chromatograms.

Copper tubing is manufactured by either a piercing or an extrusion process (References 5-9). In the piercing method, a hole is pierced through the center of a copper billet. The billet is then heated to a plastic state and rolled on a mandrel until the desired size is obtained.

In the case of the extrusion process, a billet is heated to between 650 and 900°C and placed in a preheated cylinder in a hydraulic

press. A hole is pierced in the center of the billet by a ram. A second ram forces the copper billet through an orifice in a die to form the tubing. Tremendous frictional forces must be overcome during this process. These forces are reduced by coating the chamber and the die with a lubricant that is usually made from a mixture of graphite powder and hydrocarbon grease. At the temperature involved, PAHs can be formed by pyrolysis of the lubricant.

We felt that it was necessary to determine if the contaminated tubing used in our test was an isolated instance of PAH contamination in which the tubing had not been cleaned after manufacture, or if it was a general property of all soft copper tubing.

EXPERIMENTAL

Fifty-foot sections of 1/4-inch copper tubing were obtained from Reading Industries Inc., Reading, Penn.; Halstead Metal Products Division, Zelienople, Penn.; and Mueller Brass Company, Port Huron, Mich. As supplied by the manufacturers, each section of tubing was capped, presumably to prevent contaminants from entering the tube. The tubing from Halstead Metal Products was the only sample that was labeled by the manufacturer as having been annealed. The tubing and the samples extracted from the inner surfaces are labeled R, H, and M, respectively in this report.

A 200 ml aliquot of Burdick and Jackson "Distilled in Glass" pentane was poured through each section of tubing and collected. A yellow-green fluorescence was observed in samples R and M. An aliquot of a standard solution of d_{10} -anthracene was added to each solution for use as an internal standard. The volume of each solution was reduced to 0.5 ml using a vacuum rotary evaporator. This volume reduction procedure was also carried out on a 200 ml pentane blank.

These samples were analyzed using a Hewlett-Packard 5985A gas chromatograph/mass spectrometer GC/MS in an electron ionization mode. The samples were injected through a splitless injector onto a 50 m SE-54 fused silica capillary column which was vented directly into the MS source. The oven temperature program that was used started at 0°C and proceeded to 320°C at a rate of 3°/min.

RESULTS AND DISCUSSION

A sample total ion chromatogram is presented in Figure 1. The chromatograms contained large numbers of peaks which frequently

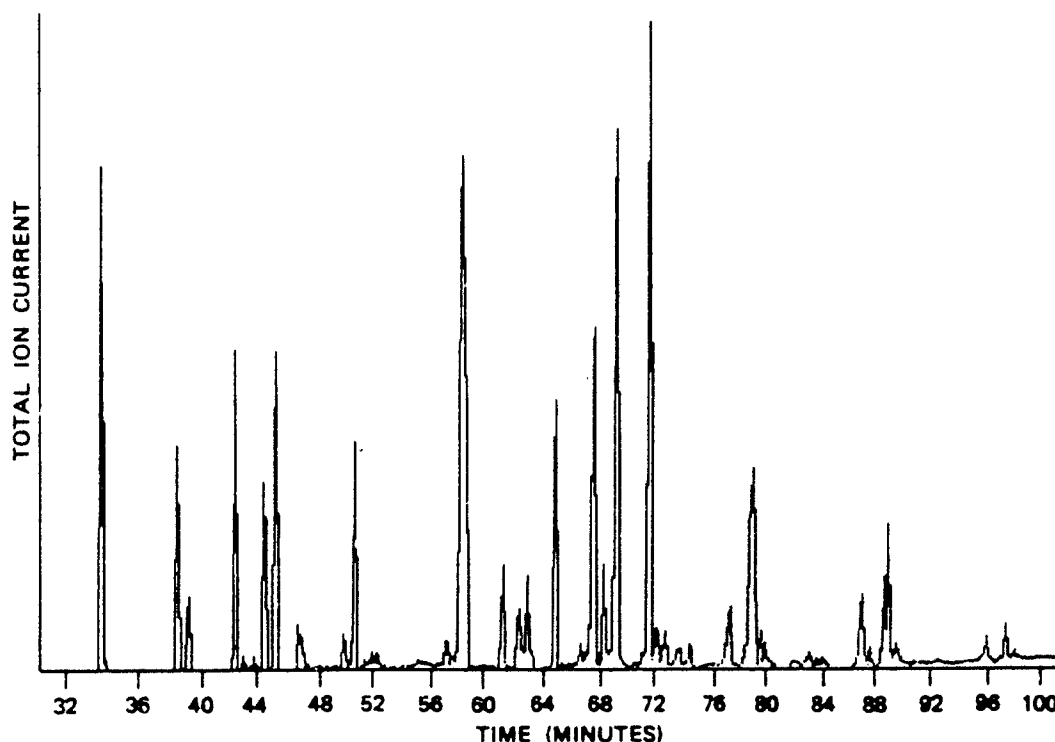


FIGURE 1. Total Ion Chromatogram of Sample R.

required the use of the mass spectral data system to separate the peaks. The data system was also used to manipulate the chromatograms in order to locate and determine the area of each peak. The internal standard, d_{10} -anthracene, was used to quantitate each peak. The contaminant compositions of each solution were used to calculate average concentration values on the inner surfaces of the copper tubing.

This method of quantitation assumes a linear response of concentration with respect to area, and it also assumes that all of the PAHs have the same response factor as the d_{10} -anthracene. The first assumption is valid for low concentrations and provides a good approximation at the levels which we observed. The second assumption is valid only for those compounds which elute in close proximity to the internal standards and for those which have a similar chemical makeup. Similar work performed in our laboratory with straight chain hydrocarbons indicate that quantities of compounds calculated by this technique will

actually be higher than those calculated for compounds which have elution times distant from the internal standard. While this quantitation method is not accurate, it does yield relative values for the concentrations of contaminants in the samples.

Retention indices were calculated using the method proposed by Lee, and others (Reference 10), which utilizes the following approximation:

$$I = 100 \frac{T_c - T_s}{T_{(s+1)} - T_s} + 100Z \quad (1)$$

where I is the retention index, T_c is the retention time of the compound of interest, T_s and $T_{(s+1)}$ are the retention times for the PAH standards which bracket the compound of interest, and Z is the number of rings in the standard which elutes just prior to the compound of interest. Standards used were naphthalene, phenanthrene, chrysene, and picene. Retention indices for compounds which were not bracketed by these standards were calculated by extending the naphthalene-phenanthrene or the chrysene-picene brackets.

Compound identification was performed by interpreting mass spectra. The electron ionization (EI) fragmentation pattern for PAHs consists of an intense molecular ion peak accompanied by smaller peaks due to the loss of one to four hydrogen atoms (Reference 11). The only other major peaks present are the doubly-charged molecular ions whose abundances are usually about 20% that of the molecular ion. The EI spectra of phthalate esters consist almost entirely of the $\text{CO}(\text{C}_6\text{H}_4)\text{COOH}^+$ fragment at an m/e value of 149 with the corresponding ^{13}C isotope at an m/e value of 150 (Reference 12). In order to positively identify phthalate esters by mass spectrometry, it is necessary to perform the analysis in a chemical ionization mode. The EI spectral information was used to obtain tentative identifications for the chromatographic peaks. In addition, the *Eight Peak Index of Mass Spectra* (Reference 13) was used to verify the identifications.

Ten oxygenated hydrocarbons and 256 PAHs were found in these three samples. Our results are listed in Appendix A. In several instances, incomplete chromatographic separation occurred in which more than one identifiable compound was found under a peak. In these cases, the compounds have been identified, but the concentration listed is for the total peak area and not for individual species. Differences in sample size and in instrumental noise resulted in different detection limits from one sample to the next.

It is not possible to identify most isomeric species by mass spectrometry. Chromatographic techniques can be used for this purpose, but they require large numbers of standards. Identification of all isomeric species present would have required a prohibitively large set of known standards and was not attempted. Molecular formulas are given in the appendices. We have identified a few of these compounds, however, and these are listed in Appendix B. We have also reported the relative carcinogenicity in this table as obtained from Futoma and others (Reference 4). These identified compounds range in relative carcinogenicity from inactive to very active. The most potent compound in this list, benzo(a)pyrene, was found in all three samples.

In determining the extent of contamination to equipment and the toxic hazards to individuals, it is important to estimate the extent of total contamination in the tubing. This was done by adding the concentration values for each sample listed in Appendix A. These data are presented in Table 1. In the case of samples R and M, the contamination is much more extensive than we would have predicted prior to this experiment. Extracts from copper tubing obtained since this work was performed, but not subjected to GC/MS analysis, have been observed to be more fluorescent than the three samples which we have analyzed. This indicates that contamination of this new tubing may be even more severe than was found with the three samples.

TABLE 1. Total Contamination
of Copper Tubing.

Sample	Total PAHs (mg/m)	Total contamination (mg/m)
R	0.17	0.18
H	0.00021	0.00069
M	0.026	0.026

CONCLUSIONS

Significant quantities of PAHs have been found in two out of three samples of copper tubing as supplied to us by the manufacturers. The exception was a sample of tubing which had been annealed. These PAHs were probably present as a result of the manufacturing process.

Since copper tubing has such a wide range of uses, this contamination should be of concern to many individuals. We recommend that new copper tubing be cleaned with suitable solvent prior to use in order to prevent contamination of equipment or personnel. In addition, we believe that data presented in this paper suggests a need to determine quantities of PAHs which can be eluted into drinking water over a period of time, and what hazard this presents to the population at large.

With such a large quantity of PAHs present in copper tubing, many of which are known carcinogens, it may be desirable for the manufacturers to market cleaned tubing for use in drinking water lines. This cleaning could be done by extracting the tubing with a solvent which leaves no toxic residues. A simple quality check could be developed using either fluorescence or ultraviolet absorbance techniques.

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Appendix A

COMPOUNDS EXTRACTED FROM COPPER TUBING

TABLE A-1. Compounds Extracted From Copper Tubing.

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
1	171.8	116	C ₉ H ₈	220.0	< 0.4	< 2.0
2	187.3	130	C ₁₀ H ₁₀	30.0	< 0.4	< 2.0
3	189.6	130	C ₁₀ H ₁₀	19.0	< 0.4	< 2.0
4	193.6	130	C ₁₀ H ₁₀	49.0	< 0.4	< 2.0
5	194.8	130	C ₁₀ H ₁₀	47.0	< 0.4	< 2.0
6	200.0	128	C ₁₀ H ₈	17,000.0	96.0	23.0
7	201.2	134	C ₁₀ H ₁₄	160.0	< 0.4	< 2.0
8	213.1	144	C ₁₁ H ₁₂	21.0	< 0.4	< 2.0
9	219.1	148	C ₁₁ H ₁₆	16.0	< 0.4	< 2.0
10	220.6	142	C ₁₁ H ₁₀	3,300.0	< 0.4	130.0
11	222.2	148	C ₁₁ H ₁₆	14.0	< 0.4	< 2.0
12	223.4	142	C ₁₁ H ₁₀	1,100.0	< 0.4	86.0
13	236.5	154	C ₁₂ H ₁₀	6,300.0	4.4	270.0
14	238.5	156	C ₁₂ H ₁₂	230.0	< 0.4	< 2.0
15	239.3	156	C ₁₂ H ₁₂	19.0	< 0.4	25.0
16	240.5	168	C ₁₃ H ₁₂	120.0	< 0.4	< 2.0
	...	156	C ₁₂ H ₁₂
17	241.1	156	C ₁₂ H ₁₂	< 10.0	< 0.4	140.0
18	241.7	154	C ₁₂ H ₁₀	230.0	< 0.4	17.0
19	242.9	156	C ₁₂ H ₁₂	51.0	< 0.4	110.0
20	243.7	156	C ₁₂ H ₁₂	22.0	< 0.4	11.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
21	244.8	154	C ₁₂ H ₁₀	3,400.0	< 0.4	170.0
22	246.0	168	C ₁₃ H ₁₂	60.0	< 0.4	64.0
23	247.2	156	C ₁₂ H ₁₂	< 10.0	< 0.4	24.0
24	248.0	152	C ₁₂ H ₈	7,600.0	< 0.4	670.0
25	248.4	156	C ₁₂ H ₁₂	21.0	< 0.4	< 2.0
26	249.4	156	C ₁₂ H ₁₂	< 10.0	< 0.4	30.0
27	252.4	168	C ₁₃ H ₁₂	18.0	< 0.4	7.7
28	254.0	154	C ₁₂ H ₁₀	740.0	< 0.4	110.0
29	254.8	168	C ₁₃ H ₁₂	520.0	< 0.4	210.0
30	256.0	168	C ₁₃ H ₁₂	240.0	< 0.4	100.0
31	257.1	156	C ₁₂ H ₁₂	21.0	< 0.4	30.0
32	258.4	182	C ₁₃ H ₁₀ ⁰	< 10.0	< 0.4	7.5
33	258.7	168	C ₁₃ H ₁₂	79.0	7.2	< 2.0
34	259.9	180	C ₁₄ H ₁₂	45.0	< 0.4	< 2.0
35	260.3	168	C ₁₃ H ₁₂	20.0	< 0.4	< 2.0
36	260.7	180	C ₁₄ H ₁₂	53.0	< 0.4	< 2.0
37	261.6	180	C ₁₄ H ₁₂	< 10.0	< 0.4	25.0
38	262.3	182	C ₁₄ H ₁₄	< 10.0	< 0.4	23.0
39	263.5	168	C ₁₃ H ₁₂	71.0	< 0.4	39.0
40	264.3	168	C ₁₃ H ₁₂	40.0	< 0.4	15.0
41	265.9	168	C ₁₃ H ₁₂	27.0	< 0.4	< 2.0
42	266.7	166	C ₁₃ H ₁₀	660.0	< 0.4	13.0

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TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
43	267.4	166	C ₁₃ H ₁₀	< 10.0	< 0.4	210.0
44	268.7	166	C ₁₃ H ₁₀	280.0	< 0.4	48.0
45	269.4	166	C ₁₃ H ₁₀	4,500.0	< 0.4	1,000.0
46	271.4	182	C ₁₄ H ₁₄	87.0	< 0.4	65.0
47	272.6	166	C ₁₃ H ₁₀	160.0	< 0.4	17.0
	...	168	C ₁₃ H ₁₂
48	273.4	182	C ₁₄ H ₁₄	50.0	< 0.4	20.0
49	273.8	168	C ₁₃ H ₁₂	31.0	< 0.4	14.0
50	274.6	180	C ₁₄ H ₁₂	340.0	< 0.4	150.0
51	275.0	166	C ₁₃ H ₁₀	120.0	< 0.4	< 2.0
52	276.2	166	C ₁₃ H ₁₀	280.0	< 0.4	< 2.0
53	277.8	182	C ₁₄ H ₁₄	< 10.0	< 0.4	16.0
54	278.2	180	C ₁₄ H ₁₂	130.0	< 0.4	11.0
55	281.4	224	C ₁₃ H ₂₀ O ₃	38.0	< 0.4	< 2.0
56	282.1	180	C ₁₄ H ₁₂	18.0	< 0.4	4.1
57	282.9	180	C ₁₄ H ₁₂	33.0	< 0.4	< 2.0
58	283.3	180	C ₁₄ H ₁₂	19.0	< 0.4	< 2.0
59	284.5	180	C ₁₄ H ₁₂	65.0	< 0.4	15.0
60	286.5	180	C ₁₄ H ₁₂	64.0	< 0.4	< 2.0
61	287.3	180	C ₁₄ H ₁₂	290.0	< 0.4	210.0
62	288.5	180	C ₁₄ H ₁₂	65.0	< 0.4	42.0
63	288.9	178	C ₁₄ H ₁₀	74.0	< 0.4	< 2.0

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TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
64	289.7	180	C ₁₄ H ₁₂	84.0	< 0.4	< 2.0
65	290.1	180	C ₁₄ H ₁₂	56.0	< 0.4	98.0
66	290.9	194	C ₁₄ H ₁₀ ^O	16.0	< 0.4	< 2.0
	...	178	C ₁₄ H ₁₀
67	292.1	192	C ₁₅ H ₁₂	40.0	< 0.4	< 2.0
68	292.9	180	C ₁₄ H ₁₂	110.0	< 0.4	86.0
69	294.1	180	C ₁₄ H ₁₂	38.0	< 0.4	< 2.0
70	294.9	184	C ₁₄ H ₁₆	660.0	< 0.4	38.0
71	300.0	178	C ₁₄ H ₁₀	23,000.0	20.0	4,000.0
72	301.0	178	C ₁₄ H ₁₀	7,800.00	2.0	1,200.0
73	302.9	180	C ₁₄ H ₁₂	41.0	< 0.4	30.0
74	303.4	184	C ₁₄ H ₁₆	40.0	< 0.4	< 2.0
75	304.8	192	C ₁₅ H ₁₂	42.0	< 0.4	< 2.0
76	305.8	192	C ₁₅ H ₁₂	35.0	< 0.4	< 2.0
77	307.2	202	C ₁₆ H ₁₀	9.7	< 0.4	< 2.0
78	308.7	192	C ₁₅ H ₁₂	32.0	< 0.4	< 2.0
79	310.6	192	C ₁₅ H ₁₂	41.0	< 0.4	33.0
80	311.5	198	C ₁₅ H ₁₈	31.0	< 0.4	< 2.0
81	313.0	204	C ₁₆ H ₁₂	1900.0	< 0.4	200.0
82	313.9	192	C ₁₅ H ₁₂	89.0	< 0.4	36.0
83	314.4	198	C ₁₅ H ₁₈	18.0	< 0.4	25.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
84	316.4	?	Phthalate Ester	18.0	< 0.4	< 2.0
85	318.3	192	C ₁₅ H ₁₂	940.0	< 0.4	880.0
86	319.2	192	C ₁₅ H ₁₂	1,100.0	< 0.4	470.0
87	320.7	192	C ₁₅ H ₁₂	350.0	< 0.4	380.0
88	321.1	190	C ₁₅ H ₁₀	< 10.0	< 0.4	570.0
89	321.6	190	C ₁₅ H ₁₀	1,900.0	< 0.4	< 2.0
90	322.1	192	C ₁₅ H ₁₂	440.0	< 0.4	350.0
91	322.6	192	C ₁₅ H ₁₂	< 10.0	< 0.4	190.0
92	323.1	192	C ₁₅ H ₁₂	310.0	< 0.4	< 2.0
93	326.4	218	C ₁₇ H ₁₄	< 10.0	< 0.4	5.7
94	326.9	218	C ₁₇ H ₁₄	25.0	< 0.4	< 2.0
95	327.4	212	C ₁₆ H ₂₀	< 10.0	< 0.4	7.6
96	328.4	192	C ₁₅ H ₁₂	29.0	< 0.4	< 2.0
	...	204	C ₁₆ H ₁₂
97	329.3	218	C ₁₇ H ₁₄	66.0	< 0.4	< 2.0
98	330.8	204	C ₁₆ H ₁₂	< 10.0	< 0.4	640.0
99	331.7	204	C ₁₆ H ₁₂	5,600.0	< 0.4	< 2.0
100	333.7	206	C ₁₆ H ₁₄	110.0	< 0.4	18.0
101	336.1	206	C ₁₆ H ₁₄	91.0	< 0.4	43.0
102	336.5	204	C ₁₆ H ₁₂	32.0	< 0.4	< 2.0
103	337.5	206	C ₁₆ H ₁₄	120.0	< 0.4	65.0
	...	218	C ₁₇ H ₁₄

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
104	339.0	204	C ₁₆ H ₁₂	380.0	< 0.4	< 2.0
105	340.4	206	C ₁₆ H ₁₄	< 10.0	< 0.4	93.0
106	340.9	206	C ₁₆ H ₁₄	36.0	< 0.4	11.0
107	341.8	206	C ₁₆ H ₁₄	< 10.0	< 0.4	4.8
108	342.3	204	C ₁₆ H ₁₂	540.0	< 0.4	6.4
109	344.7	202	C ₁₆ H ₁₀	11,000.0	9.1	1,200.0
110	348.1	202	C ₁₆ H ₁₀	2,200.0	0.41	360.0
111	349.5	202	C ₁₆ H ₁₀	230.0	< 0.4	< 2.0
	...	208	C ₁₆ H ₁₆
112	351.9	202	C ₁₆ H ₁₀	< 10.0	< 0.4	2,700.0
113	352.9	202	C ₁₆ H ₁₀	20,000.0	11.0	54.0
114	353.4	218	C ₁₇ H ₁₄	230.0	< 0.4	< 2.0
115	354.3	204	C ₁₆ H ₁₂	73.0	< 0.4	< 2.0
116	357.2	202	C ₁₆ H ₁₀	18.0	< 0.4	< 2.0
	...	242	C ₁₉ H ₁₄
117	358.2	230	C ₁₈ H ₁₄	120.0	< 0.4	8.2
118	358.7	220	C ₁₇ H ₁₆	< 10.0	< 0.4	3.0
119	363.0	216	C ₁₇ H ₁₂	580.0	< 0.4	190.0
120	363.9	314	C ₁₈ H ₃₄ O ₄	15,000.0	460.0	82.0
121	365.9	216	C ₁₇ H ₁₂	< 10.0	< 0.4	420.0
122	366.4	216	C ₁₇ H ₁₂	1,300.0	1.1	21.0
123	367.8	230	C ₁₈ H ₁₄	42.0	< 0.4	< 2.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
124	368.8	216	C ₁₇ H ₁₂	1,100.0	< 0.4	< 2.0
125	369.2	216	C ₁₇ H ₁₂	< 10.0	< 0.4	580.0
126	369.7	216	C ₁₇ H ₁₂	780.0	3.3	< 2.0
127	370.2	230	C ₁₈ H ₁₄	21.0	< 0.4	< 2.0
128	371.2	218	C ₁₇ H ₁₄	42.0	< 0.4	5.2
	...	230	C ₁₈ H ₁₄
129	372.6	216	C ₁₇ H ₁₂	420.0	< 0.4	< 2.0
130	373.1	216	C ₁₇ H ₁₂	< 10.0	< 0.4	250.0
131	374.0	216	C ₁₇ H ₁₂	370.0	< 0.4	140.0
132	375.0	228	C ₁₈ H ₁₂	370.0	< 0.4	22.0
133	376.4	230	C ₁₈ H ₁₄	34.0	< 0.4	< 2.0
134	377.4	328	C ₁₉ H ₃₆ O ₄	440.0	< 0.4	< 2.0
135	379.3	230	C ₁₈ H ₁₄	22.0	< 0.4	9.8
136	380.3	230	C ₁₈ H ₁₄	38.0	< 0.4	< 2.0
137	381.7	242	C ₁₉ H ₁₄	120.0	< 0.4	< 2.0
138	383.2	228	C ₁₈ H ₁₂	120.0	< 0.4	64.0
139	383.7	230	C ₁₈ H ₁₄	73.0	< 0.4	< 2.0
140	385.1	230	C ₁₈ H ₁₄	20.0	< 0.4	< 2.0
141	385.6	230	C ₁₈ H ₁₄	21.0	< 0.4	90.0
142	387.0	?	Phthalate Ester	93.0	< 0.4	< 2.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
143	388.9	228	C ₁₈ H ₁₂	340.0	< 0.4	35.0
	...	234	C ₁₈ H ₁₈
144	390.4	226	C ₁₈ H ₁₀	1,300.0	< 0.4	140.0
145	390.9	228	C ₁₈ H ₁₂	1,100.0	< 0.4	120.0
146	392.3	234	C ₁₈ H ₁₈	54.0	< 0.4	6.3
147	393.3	230	C ₁₈ H ₁₄	< 10.0	< 0.4	58.0
148	393.8	242	C ₁₉ H ₁₄	28.0	< 0.4	< 2.0
149	395.2	234	C ₁₈ H ₁₈	95.0	< 0.4	12.0
	...	228	C ₁₈ H ₁₂
	...	254	C ₂₀ H ₁₄
150	397.1	226	C ₁₈ H ₁₀	590.0	< 0.4	< 2.0
151	398.1	228	C ₁₈ H ₁₂	< 10.0	< 0.4	650.0
152	398.6	228	C ₁₈ H ₁₂	4,400.0	5.7	< 2.0
153	400.0	228	C ₁₈ H ₁₂	4,600.0	12.0	600.0
154	402.2	228	C ₁₈ H ₁₂	630.0	< 0.4	< 2.0
155	402.8	242	C ₁₉ H ₁₄	< 10.0	< 0.4	97.0
156	404.5	254	C ₂₀ H ₁₄	700.0	< 0.4	21.0
157	405.6	230	C ₁₈ H ₁₄	< 10.0	< 0.4	280.0
158	406.2	254	C ₂₀ H ₁₄	110.0	< 0.4	12.0
159	407.3	254	C ₂₀ H ₁₄	< 10.0	< 0.4	11.0
160	407.9	242	C ₁₉ H ₁₄	< 10.0	< 0.4	19.0
161	408.4	254	C ₂₀ H ₁₄	100.0	< 0.4	< 2.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
162	411.2	242	C ₁₉ H ₁₄	< 10.0	< 0.4	14.0
163	414.5	?	Phthalate Ester	34.0	12.0	< 2.0
164	415.7	242	C ₁₉ H ₁₄	160.0	1.6	62.0
	...	?	Phthalate Ester
165	417.4	242	C ₁₈ H ₁₄	200.0	< 0.4	190.0
166	418.5	242	C ₁₉ H ₁₄	65.0	< 0.4	67.0
167	420.2	240	C ₁₉ H ₁₂	110.0	< 0.4	< 2.0
168	420.8	242	C ₁₉ H ₁₄	< 10.0	< 0.4	19.0
169	421.3	240	C ₁₉ H ₁₂	72.0	< 0.4	< 2.0
170	422.5	242	C ₁₉ H ₁₄	< 10.0	< 0.4	120.0
	...	254	C ₂₀ H ₁₄
171	423.1	254	C ₂₀ H ₁₄	450.0	< 0.4	< 2.0
172	424.7	254	C ₂₀ H ₁₄	< 10.0	< 0.4	25.0
173	425.3	254	C ₂₀ H ₁₄	250.0	< 0.4	< 2.0
174	427.5	254	C ₂₀ H ₁₄	< 10.0	< 0.4	32.0
175	428.1	254	C ₂₀ H ₁₄	260.0	< 0.4	< 2.0
176	429.2	254	C ₂₀ H ₁₄	130.0	< 0.4	< 2.0
177	429.8	254	C ₂₀ H ₁₄	< 10.0	< 0.4	13.0
178	433.7	256	C ₂₀ H ₁₆	< 10.0	< 0.4	15.0
179	436.5	254	C ₂₀ H ₁₄	< 10.0	< 0.4	9.8

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
180	438.8	256	C ₂₀ H ₁₆	< 10.0	< 0.4	8.9
	...	266	C ₂₁ H ₁₄
181	442.7	252	C ₂₀ H ₁₂	< 10.0	< 0.4	500.0
182	443.8	252	C ₂₀ H ₁₂	3,800.0	12.0	< 2.0
183	447.2	252	C ₂₀ H ₁₂	450.0	< 0.4	68.0
184	449.9	280	C ₂₂ H ₁₆	33.0	< 0.4	< 2.0
185	453.4	252	C ₂₀ H ₁₂	2,600.0	10.0	560.0
186	455.0	252	C ₂₀ H ₁₂	3,700.0	16.0	560.0
187	457.3	252	C ₂₀ H ₁₂	< 10.0	< 0.4	120.0
188	457.8	252	C ₂₀ H ₁₂	560.0	< 0.4	< 2.0
189	458.4	278	C ₂₂ H ₁₄	< 10.0	< 0.4	18.0
190	459.5	278	C ₂₂ H ₁₄	160.0	< 0.4	< 2.0
191	460.1	266	C ₂₁ H ₁₄	< 10.0	< 0.4	120.0
192	460.7	266	C ₂₁ H ₁₄	42.0	< 0.4	< 2.0
193	464.6	266	C ₂₁ H ₁₄	< 10.0	< 0.4	93.0
	...	278	C ₂₂ H ₁₄
194	465.1	278	C ₂₂ H ₁₄	88.0	< 0.4	< 2.0
195	465.7	266	C ₂₁ H ₁₄	34.0	< 0.4	< 2.0
196	468.5	266	C ₂₁ H ₁₄	200.0	< 0.4	98.0
	...	278	C ₂₂ H ₁₄
197	470.8	266	C ₂₁ H ₁₄	< 10.0	< 0.4	55.0

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
198	473.0	264	C ₂₁ H ₁₂	66.0	< 0.4	68.0
	...	266	C ₂₁ H ₁₄
199	474.7	278	C ₂₂ H ₁₄	< 10.0	< 0.4	22.0
200	475.3	278	C ₂₂ H ₁₄	98.0	< 0.4	< 2.0
201	483.7	278	C ₂₂ H ₁₄	72.0	< 0.4	9.2
202	486.0	278	C ₂₂ H ₁₄	< 10.0	< 0.4	14.0
203	486.6	278	C ₂₂ H ₁₄	120.0	< 0.4	< 2.0
204	487.6	278	C ₂₂ H ₁₄	< 10.0	< 0.4	17.0
205	488.7	278	C ₂₂ H ₁₄	46.0	< 0.4	< 2.0
206	491.0	278	C ₂₂ H ₁₄	240.0	< 0.4	40.0
	...	276	C ₂₂ H ₁₂
207	493.3	276	C ₂₂ H ₁₂	< 10.0	1.1	150.0
208	493.9	276	C ₂₂ H ₁₂	650.0	< 0.4	< 2.0
209	494.4	278	C ₂₂ H ₁₄	< 10.0	< 0.4	41.0
210	495.5	278	C ₂₂ H ₁₄	91.0	< 0.4	< 2.0
211	496.1	278	C ₂₂ H ₁₄	< 10.0	< 0.4	7.6
212	496.7	278	C ₂₂ H ₁₄	15.0	< 0.4	< 2.0
213	497.8	278	C ₂₂ H ₁₄	< 10.0	< 0.4	88.0
214	498.3	278	C ₂₂ H ₁₄	92.0	< 0.4	< 2.0
215	499.4	278	C ₂₂ H ₁₄	53.0	< 0.4	< 2.0
216	501.1	276	C ₂₂ H ₁₂	810.0	3.4	230.0
217	502.8	278	C ₂₂ H ₁₄	< 10.0	< 0.4	4.8

TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
218	503.3	278	C ₂₂ H ₁₄	25.0	< 0.4	< 2.0
219	504.0	292	C ₂₃ H ₁₆	2.5	< 0.4	< 2.0
220	504.5	276	C ₂₂ H ₁₂	250.0	< 0.4	10.0
221	506.2	290	C ₂₃ H ₁₄	< 10.0	< 0.4	9.5
222	507.3	292	C ₂₃ H ₁₆	< 10.0	< 0.4	3.6
223	508.4	290	C ₂₃ H ₁₄	< 10.0	< 0.4	12.0
224	509.0	290	C ₂₃ H ₁₄	11.0	< 0.4	< 2.0
225	509.6	290	C ₂₃ H ₁₄	52.0	< 0.4	< 2.0
226	510.7	292	C ₂₃ H ₁₆	< 10.0	< 0.4	9.7
227	512.4	290	C ₂₃ H ₁₄	< 10.0	< 0.4	7.4
228	514.0	?	Phthalate Ester	72.0	< 0.4	22.0
229	514.6	290	C ₂₃ H ₁₄	80.0	< 0.4	31.0
230	515.2	290	C ₂₃ H ₁₄	17.0	< 0.4	< 2.0
231	518.0	290	C ₂₃ H ₁₄	6.2	< 0.4	< 2.0
232	520.2	290	C ₂₃ H ₁₄	20.0	< 0.4	31.0
233	534.8	302	C ₂₄ H ₁₄	150.0	< 0.4	54.0
234	535.9	302	C ₂₄ H ₁₄	94.0	< 0.4	< 2.0
235	536.5	302	C ₂₄ H ₁₄	< 10.0	< 0.4	50.0
236	537.1	302	C ₂₄ H ₁₄	140.0	< 0.4	< 2.0
237	542.7	302	C ₂₄ H ₁₄	34.0	< 0.4	11.0
238	544.9	300	C ₂₄ H ₁₂	52.0	< 0.4	37.0
239	546.1	302	C ₂₄ H ₁₂	160.0	< 0.4	95.0

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TABLE A-1. (Contd.)

Peak number	Retention index	Molecular weight	Molecular formula	Concentration (ng/m)		
				R	H	M
240	547.8	302	C ₂₄ H ₁₂	< 10.0	< 0.4	30.0
241	548.4	302	C ₂₄ H ₁₂	34.0	< 0.4	< 2.0
242	551.7	316	C ₂₅ H ₁₆	< 10.0	< 0.4	21.0
243	555.1	316	C ₂₅ H ₁₆	< 10.0	< 0.4	12.0
244	559.0	316	C ₂₅ H ₁₆	< 10.0	< 0.4	37.0
245	574.7	326	C ₂₆ H ₁₄	< 10.0	< 0.4	5.9
246	585.4	326	C ₂₆ H ₁₄	< 10.0	< 0.4	16.0
247	594.4	326	C ₂₆ H ₁₄	< 10.0	< 0.4	8.2
248	596.6	326	C ₂₆ H ₁₄	< 10.0	< 0.4	20.0
249	607.9	326	C ₂₆ H ₁₄	< 10.0	< 0.4	2.6

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Appendix B

IDENTIFICATION OF CHROMATOGRAPHIC PEAKS

TABLE B-1. Identification of Chromatographic Peaks.

Peak number	Identification	Relative ^a carcinogenicity (4)
6	Naphthalene	-
13	Biphenyl	-
24	Acenaphthylene	...
28	Acenaphthene	-
32	Fluorenol	...
45	Fluorene	-
71	Phenanthrene	-
72	Anthracene	-
109	Fluoroanthene	-
113	Pyrene	-
120	Dibutyl sebecate	...
152	Benzo(a)anthracene	<+
153	Chrysene	<+
181	Benzo(b)fluoranthene	++
182	Benzo(k)fluoranthene	-
185	Benzo(e)pyrene	<+
186	Benzo(a)pyrene	++++
208	Indeno(1,2,3-c,d)pyrene	+
210	Dibenzo(a,h)anthracene	+++
216	Benzo(g,h,i)perylene	-

^a++++ = very active, +++ = active, ++ = moderately active, + = weakly active, - = inactive or unknown.

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 - AFATL/DLODL (1)
 - D. Harrison (1)
- 1 Air Force Occupational and Environmental Health Laboratory, Brooks Air Force Base (AFOEHL/SA)
- 1 Air Force School of Aerospace Medicine, Brooks Air Force Base (Crew Technology Division)
- 2 Air Force Wright Aeronautical Laboratory, Wright-Patterson Air Force Base
 - AFWAL/MLLM (1)
 - AFWAL/MLTM (1)
- 12 Defense Technical Information Center
 - 1 Department of the Treasury, Bureau of Alcohol, Tobacco & Firearms (Forensic Science Branch, R. A. Strobel)
 - 1 Battelle Memorial Laboratory, Tactical Technology Information Center, Columbus, OH
 - 1 Holston Defense Corporation, Kingsport, TN (Plant Manager)
 - 1 Los Alamos National Laboratory, Los Alamos, NM (H. Flaugh)
 - 1 Los Angeles Police Department, Los Angeles, CA (Scientific Investigation Division, C-8087, B. Sanchez)
 - 3 The Johns Hopkins University, Applied Physics Laboratory, Chemical Propulsion Information Agency, Laurel, MD
 - T. W. Christian (2)
 - J. Hannum (1)
 - 2 University of California, Lawrence Livermore National Laboratory, Livermore, CA Organic Materials Division
 - R. R. McGuire (1)
 - D. L. Ornellas (1)
 - 1 University of Connecticut, Storrs, CT (Chemistry Department, S. R. Smith)